National Defence / Défense nationale

OBSERVATIONS ON THE USE OF THE FTS 400 (U)

by

J.R. Coleman
Chemical Protection Section
Protective Sciences Division

DEFENCE RESEARCH ESTABLISHMENT OTTAWA
TECHNICAL NOTE 87-19

PCN
051LB13

May 1987
Ottawa

ABSTRACT

A filter test system, the FTS 400, is capable of generating monodisperse aerosols in the size range 0.01 to 10 μm. This note describes operation of the main instrumental components, and notes precautions necessary in interpretation of results.

RÉSUMÉ

Un système d'essai des filtres, le FTS 400, est capable de produire des aérosols monodispersés dans la plage de diamétres de particules de 0,01 à 10 μm. La présente notice décrit le fonctionnement des principales composantes de l'instrumentation et souligne les précautions à prendre dans l'interprétation des résultats.

TABLE OF CONTENTS

		Page
ABSTRACT/RÉSUMÉ		(iii)
1.0	INTRODUCTION	1
2.0	PROCEDURES AND DESCRIPTION OF APPARATUS	1
2.1	Production of Aerosol	1
2.2	Condensation Nucleus Counter	3
2.3	Aerodynamic Particle Sizer	3
2.4	Electrostatic Classifier	4
2.5	Diagnostics for Classifier Operation	7
2.6	Aerosol Concentrations	8
3.0	CONCLUDING OBSERVATIONS	9
4.0	BIBLIOGRAPHY	9
APPENDIX	- DAILY CHECKOUT OF FTS 400	11

1.0 INTRODUCTION

This note contains remarks on the use of the FTS 400, a system designed by TSI Inc. to meet DREO requirements in filter testing. The instruction manual (1) provided by the company, and the individual manuals for the component instruments, are primary sources of information. This note is intended only to supplement these manuals, and to offer practical suggestions on day to day operation and maintenance to persons with no previous acquaintance with the system.

A schematic, taken from the manual, appears as Figure 1. The major instrumental components are the electrostatic classifier (TSI 3071), condensation nucleus counter or CNC (TSI 3020) and aerodynamic particle sizer or APS (TSI APS 3300). Operation of the three instruments and of auxiliary equipment (atomizers, transducer, etc) is controlled by a program written for the IBM PC. This program takes the system through a test procedure with parameters keyed in by the operator, stores and performs necessary manipulations on the test data, and displays and prints out the results. Detailed instruction on capabilities, use and servicing of these (and of other minor components of the system) are included as sections in the manual. A checkout procedure for day-to-day operation of the DREO system is given in the Appendix.

In summary a monodisperse aerosol is produced, diluted with filtered air and passed through the test object. Concentrations upstream and downstream are measured and filter efficiency is calculated.

2.0 PROCEDURE AND DESCRIPTION OF APPARATUS

2.1 Production of Aerosol

In the range 0.01 - 0.30 μm diameter, polydisperse aerosols are produced by atomizing and drying solutions of sodium chloride in water or dioctyl phthalate in isopropanol. The electrostatic classifier selects a narrowly defined size in the range quoted above, which is then employed as the challenge. Concentrations are measured using the CNC, whose principle of operation is described below.

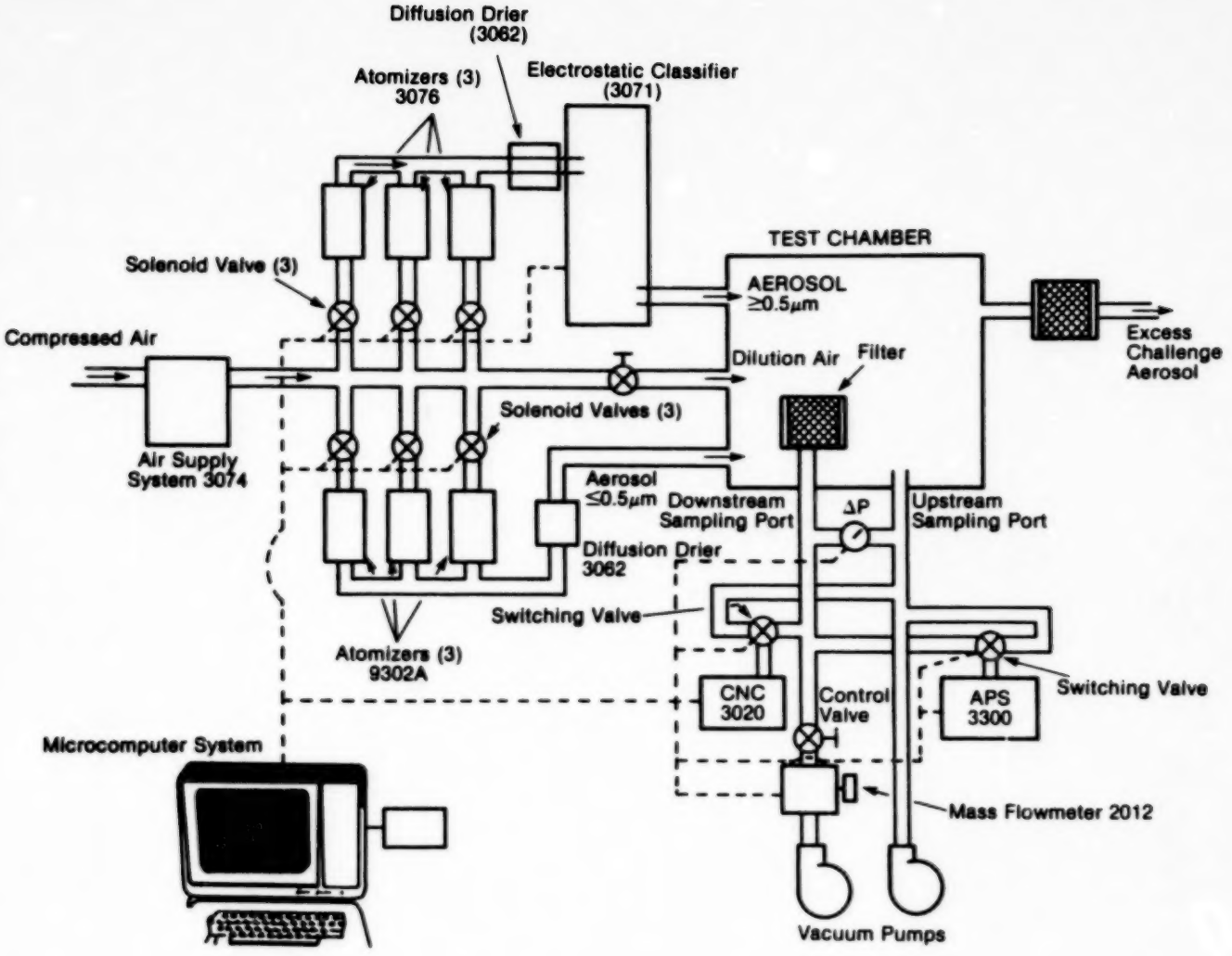

Figure 1: Schematic of FTS 400.

While the classifier is capable, operating as a standalone instrument, of selecting out particles as large as 1.0 μm, operational compromises in the FTS system, related probably to air flow through the classifier, limit the upper size to 0.31 μm.

For particle size > 0.5 μm, the aerosol source is aqueous suspensions of monodisperse (very uniformly sized) polystyrene latex spheres (PSL), stored in the 9302A atomizers of Figure 1. A suspension is dried and diluted and passed through the test object. For reasons given below the APS and not the CNC must be used as the count instrument.

2.2 Condensation Nucleus Counter

In the CNC, the sampled aerosol stream is saturated with butanol vapor at 35°C. On subsequent cooling the butanol, now supersaturated, condenses on every particle in the stream, and regardless of original size, shape or chemical nature, grows them all to butanol droplets of a uniform final diameter of 12 μm. Number concentrations in this uniform suspension of liquid droplets are determined by optical means.

Polystyrene latex spheres are purchased as suspensions of 10% weight content, which are diluted down with water for use in the atomizers. Surfactants have been added to maintain the suspension and prevent coagulation. When the diluted suspension is atomized the resulting droplets may contain 0, 1, 2 or a larger number of spheres. In order to minimize the formation of doublets, triplets, etc, the solutions are diluted down to such an extent that most of the droplets formed on atomization are "empty", i.e. have no PSL spheres, a small fraction contain one sphere, a still smaller fraction two, etc. When the suspension is dried, however, even the supposedly empty droplets leave a small residue of surfactant, and all these, along with the PSL spheres themselves will be grown in the CNC to butanol droplets and counted. Thus the CNC readings are meaningless, being typically 50-100 times the correct value determined by the APS.

2.3 Aerodynamic Particle Sizer

The principle of operation of the APS has been described elsewhere (2). The instrument was developed for characterizing aerosols, and provides in the first instance a number distribution versus size

(expressed as aerodynamic diameter) and from this a number of more complex distribution measurements of interest in aerosol investigations. When under FTS 400 program control only a small part of this capability is used. The APS, sampling upstream, locates the peak corresponding to the PSL, at a size significantly larger than the background residues, which are present in much larger number concentration. Having located and performed a number count on PSL upstream, it then measures the (usually considerably smaller) concentration in the same size interval downstream.

There are four instrument settings (photomultiplier tube, nozzle pressure drop (ΔP), sheath air and total air flow) whose values, with their tolerances are given in the FTS manual. Griffiths et al (3) state that the most critical adjustment is nozzle ΔP, next in importance sheath air and finally total air flow. Sheath air can be varied widely without much effect on the other two. Nozzle ΔP is extremely sensitive to small setting changes in total air flow; this latter control is then used to set ΔP precisely, and one accepts whatever total flow setting results when this is done. Depending on the condition of the filters in the APS total flow can run as much as 20 - 25 mV high (mass flowmeter voltage output) but this is not serious.

The APS has been used when the monodisperse source was PSL spheres in the size range 0.3 - 0.5 μm i.e., below the stated lower range of the instrument. The resulting number concentrations appear satisfactory from internal evidence; however the sizes measured by the APS (> 0.5 μm) are incorrect and can be ignored.

2.4 Electrostatic Classifier

The atomizers that provide polydisperse aerosol as input to the classifier produce a suspension of droplets whose size distribution is representable roughly as a log normal curve (Figure 2). On removal of solvent an aerosol of crystals (NaCl) or droplets (DOP) will be left, with the same sort of distribution. Passage through a neutralizer reduces the original random charge distribution to a Boltzmann equilibrium, which for various particle sizes has been calculated as in Table I (reference 4).

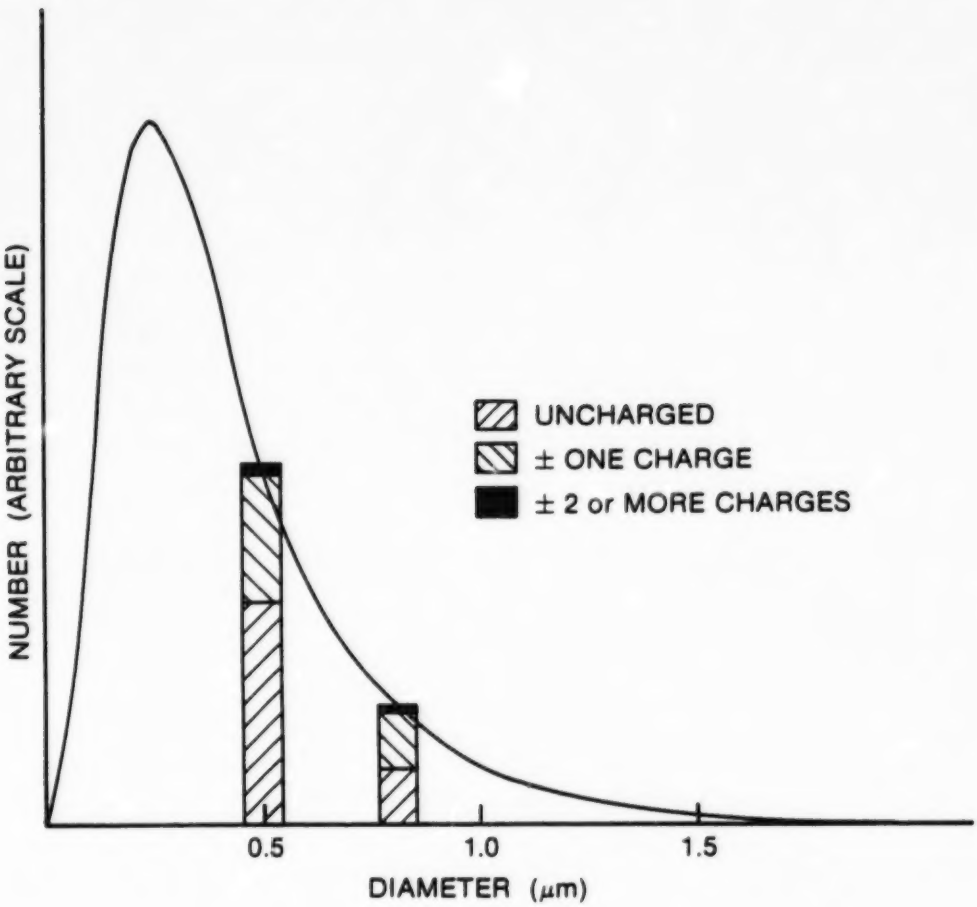

Figure 2: Representation of charge distribution of charge-equilibrated aerosol at two sizes. Distribution curve is schematic.

TABLE I

Percent of particles carrying n_p elementary charge units

Particle Diameter (μm)	n_p=-4	-3	-2	-1	0	+1	+2	+3	+4
0.01				0.34	99.32	0.34			
0.02				5.23	89.53	5.23			
0.04			0.23	16.22	67.10	16.22	0.23		
0.06		0.01	1.25	21.30	54.88	21.30	1.25	0.01	
0.08		0.08	2.78	23.37	47.53	23.37	2.78	0.08	
0.10		0.26	4.39	24.09	42.52	24.09	4.39	0.26	
0.20	0.32	2.33	9.66	22.63	30.06	22.63	9.66	2.33	0.32
0.40	2.19	5.92	12.05	18.44	21.26	18.44	12.05	5.92	2.19
0.60	3.82	7.41	11.89	15.79	17.36	15.79	11.89	7.41	3.82
0.80	4.83	7.94	11.32	14.00	15.03	14.00	11.32	7.94	4.83
1.00	5.42	8.06	10.71	12.70	13.45	12.70	10.71	8.06	5.42

The classifier resembles a cylindrical condenser with provision for introduction and withdrawal of several air and aerosol streams. An adjustable potential is maintained between the inner and outer cylinders, and uniform laminar air flows are established through the classifier, so that from the inital polydisperse aerosol a fraction of very closely defined mobility range is segregated, whose value depends on air flows, condenser geometry and potential difference. The monotonic relationship between mobility and Stokes law diameter ensures that this separated fraction lies in a correspondingly narrow range of diameters.

Only an extremely small fraction of the original heterogeneous aerosol is used. Number distribution as a function of size will, as noted above, be represented by the curve of Figure 2. When this is passed through the neutralizer each size will independently assume its own Boltzmann distribution of charge. If, then, a NaCl aerosol of 0.05 μm diameter is desired this would be represented schematically by the narrow vertical bar in Figure 2; and only about 20% of this, the fraction possessing a +1 charge at equilibrium, will be finally selected.

It can be demonstrated, using the Stokes equation, that a doubly charged particle 0.083 μm in diameter possesses the same mobility as a singly charged 0.05 μm particle, and both will exit the classifier together. To minimize this interference, three concentrations of NaCl (0.01, 0.1 and 1% w/w) are used; these divide up the size range into three domains (0.01 to <0.02, 0.02 to <0.10, and 0.10 to 0.31 μm). The intention is, as far as possible, to provide in each size range a solution concentration such that the chosen size will lie, as in Figure 2, on the

right hand, falling side of the curve, so that interference (in this case of 0.083 μm particles with 0.05 μm particles) will be kept small. It should be observed that the log-normal curve depicted in Figure 2 was chosen for illustrative purposes, and probably will not represent the actual output of the three model 3076 atomizers used in the system. Figure 2 is intended only to demonstrate the objective of the experimental arrangements.

In generating DOP aerosols from solutions in isopropanol, the high volatility of the latter makes it necessary to discard solutions after passage through the atomizer, rather than recirculating them back to stock which would thus be progressively concentrated. Two solutions (0.05 and 0.2 % w/w) are used, covering the size range 0.01 to 0.05, and >0.05 to 0.31 μm; the third bottle is used to catch drainings from the atomizers.

2.5 Diagnostics for Classifier Operation

When the electrostatic classifier is performing properly the upstream concentrations of NaCl and DOP are quite constant, varying by only a few percent from run to run, and not drifting by more than 20 - 30% over a period of weeks. Concentrations to be expected (at 32 LPM) for various size ranges are listed below, in particles/cm^3.

Diameter μm	0.01	0.02	0.05	0.10	0.20	0.30
Conc NaCl	$2\text{-}2\times10^3$	$3\text{-}5\times10^4$	$1\text{-}2\times10^5$	$2\text{-}4\times10^5$	$6\text{-}9\times10^4$	$2\text{-}4\times10^4$
DOP	$1\text{-}5\times10^2$	$2\text{-}4\times10^3$	$6\text{-}9\times10^4$	3×10^5	8×10^4	3×10^4

The wide variations at 0.01 μm are probably due to the fact that the instrument is operating at its lower limit.

The ability to produce such concentrations is a useful indication of the proper functioning of the system. When used in the FTS the major air flows entering and leaving the CNC, i.e. sheath and excess air flows are set at 10 LPM. Sheath air is adjusted directly, but excess air is controlled by varying the significantly smaller monodisperse aerosol output. The more this is reduced the greater is the excess air flow. In effect control of a small output is used to regulate a larger output, and this affords a sensitive way to test when the filter on the excess air line is becoming clogged. With a fresh filter in the line, the monodisperse control has to be opened fairly wide (- 6 full turns from the closed

position) to adjust excess flow to 10 LPM; and this valve position will remain nearly unchanged until the onset of clogging in the excess air line filter. Then it will suddenly become necessary to cut monodisperse aerosol output down, by closing the valve, in order to maintain excess air flow at 10 LPM. At this point the excess air filter must be changed.

2.6 Aerosol Concentrations

With an upstream concentration of 10^4 particles/1 c.c. of an aerosol 0.1 μm in diameter and of unit density, it is readily shown that 0.17 μg of aerosol is delivered to the test object per minute. During a test the total amount is of course directly proportional to run time, flow rate, particle density and number concentration, and to the cube of the diameter.

From the figures given above for upstream concentrations of NaCl and DOP generated, the loadings in μg/min at 32 LPM for a number of sizes are of this order:

Diameter(μm)	0.01	0.02	0.05	0.10	0.20	0.30
NaCl	.000007- .00007	.01	.7	10	20	30
DOP	.000005	.0004	.15	3	10	15

With PSL spheres of the two sizes found most useful the corresponding figures are:

Diameter μm	0.56	0.91
Upstream conc	$5\text{-}8\times10^2$	$1\text{-}2\times10^3$
Loading μg/min at 32 LPM	2	20

These concentrations are considerably lower than those generated in say the Q-127 test. However, problems may arise in testing high efficiency filters, in which downstream concentrations are exceedingly

small, especially at the extremes of size range, 0.01-0.02 μm and >0.5 μm. The measuring system, CNC or APS, counts individual particles, and when the count descends to numbers of the order of ten, the uncertainty is very great. This must be compensated for by setting long sampling times, perhaps 30 - 60 minutes, and if one is considering using the filter in subsequent tests, it may be desirable to calculate the extent of loading that occurs during this time.

An empirical check on the condition of a filter which is undergoing a series of tests is to determine efficiency periodically with 0.02 μm or 0.05 μm NaCl and see whether it has changed.

It should be noted that the operator sets for each particle size a maximum downstream sampling time, and the instrument samples either up to that maximum time, or until the concentration calculated from a periodically averaged running count satisfies statistical criteria built into the program, at which point it performs an efficiency calculation for that size and proceeds to the next size. Thus a good filter can take appreciably longer to test than a poor one.

3.0 CONCLUDING OBSERVATIONS

The FTS 400 will conduct repetitive, routine filter testing with little attention. It is necessary to make slight adjustments to the classifier flows when the system switches from one atomizer to the next.

The APS pump, though not the laser, must be switched on, to maintain downstream flows, even when no measurements are being taken in the micron range. Slight clogging in filters and nozzles results in very erratic air flows in the instrument. It may thus be necessary, after extensive work in the submicron range, to change and clean APS filters and nozzles before resuming work at larger particle sizes.

4.0 BIBLIOGRAPHY

1. TSI FTS 400 Filter Test System, Operator's Manual.

(2) Remiarz, R.J., Agarwal, J.K., Quant, F.R., Sem, G.J. A real-time aerodynamic particle size analyzer, in "Aerosols in the Mining and Industrial Work Environment" V.A. Marple and B.Y.H. Liu, Editors, Ann Arbor Sciences, Michigan, pp 879 - 895 (1983).

(3) Griffiths, W.D., Iles, P.J., and Vaughan, N.P. 1, 3 - 9, (1986).

(4) Pui, D.Y.H., and Liu, B.Y.H., Electrical Aerosol Analyzer: Calibration and Performance, in "Aerosol Measurement" D.A. Lundgren et al, editors, University Presses of Florida, Gainesville, FL, pp 384-399 (1979).

APPENDIX

DAILY CHECKOUT OF FTS 400

At start of day:

(1) Turn on compressor.

(2) Turn on systems and instrument power. Turn on APS, CNC, classifier (if APS is not to be used, the laser need not be switched on, but pump should be turned on to maintain air flow in system).

(3) Drain and refill butanol in CNC.

(4) Check main air supply desiccant. Progress of exhaustion from bottom to top of tubes is apparent.

(5) Remix desiccant in diffusion driers. Desiccant is used only in immediate vicinity of the screen tube that passes down the centre of the drier. This is seen as a white streak when the desiccant is poured out into a beaker, but is not visible from the outside. When the entire mass of desiccant has begun to fade from deep to light blue, it should be regenerated.

(6) Prepare or resuspend PSL suspensions. Prepare NaCl or DOP solutions as required.

(7) Admit air to system. (Butanol drain and refill in CNC should be complete.) It is important that systems be switched on and a program be in control before admission of air. It has been observed, during some system modifications, that when air was admitted with the switches "System Power" and "Instrument Power" ON, but with the computer out of the system so that no program was operating, all four PSL atomizers were activated. This will lead to swamping of the PSL diffusion driers.

(8) Check APS flow rates and PMT readings, if APS is to be used, (allow 20 minutes warmup).

(9) Adjust classifier settings. Note whether monodisperse aerosol valve is in same position, or has to be closed to get correct excess air flow.

(10) Follow prompts to start test.

At end of use:

(1) Cut off air supply to system.
(2) Shut down system and instrument power.
(3) Shut off compressor, drain compressor tank.
(4) Drain water traps in main air supply system and in diffusion driers.
(5) Drain manifolds (2) downstream from atomizers.

DOCUMENT CONTROL DATA

(Security classification of title, body of abstract and indexing annotation must be entered when the overall document is classified)

1. ORIGINATOR (the name and address of the organization preparing the document. Organizations for whom the document was prepared, e.g. Establishment sponsoring a contractor's report, or tasking agency, are entered in section 8.)

DEFENCE RESEARCH ESTABLISHMENT OTTAWA
Department of National Defence
Ottawa, Ontario K1A 0Z4

2. SECURITY CLASSIFICATION (overall security classification of the document. including special warning terms if applicable)

UNCLASSIFIED

3. TITLE (the complete document title as indicated on the title page. Its classification should be indicated by the appropriate abbreviation (S,C,R or U) in parentheses after the title.)

OBSERVATIONS ON THE USE OF THE FTS 400 (U)

4. AUTHORS (Last name, first name, middle initial. If military, show rank, e.g. Doe, Maj. John E.)

COLEMAN, John R.

5. DATE OF PUBLICATION (month and year of publication of document)

MAY 1987

6a. NO. OF PAGES (total containing information. Include Annexes, Appendices, etc.)

15

6b. NO. OF REFS (total cited in document)

4

7. DESCRIPTIVE NOTES (the category of the document, e.g. technical report, technical note or memorandum. If appropriate, enter the type of report, e.g. interim, progress, summary, annual or final. Give the inclusive dates when a specific reporting period is covered.)

TECHNICAL NOTE

8. SPONSORING ACTIVITY (the name of the department project office or laboratory sponsoring the research and development. Include the address.)

DREO

9a. PROJECT OR GRANT NO. (if appropriate, the applicable research and development project or grant number under which the document was written. Please specify whether project or grant)

051LB13

9b. CONTRACT NO. (if appropriate, the applicable number under which the document was written)

10a. ORIGINATOR'S DOCUMENT NUMBER (the official document number by which the document is identified by the originating activity. This number must be unique to this document.)

DREO TECHNICAL NOTE NO. 87-19

10b. OTHER DOCUMENT NOS. (Any other numbers which may be assigned this document either by the originator or by the sponsor)

11. DOCUMENT AVAILABILITY (any limitations on further dissemination of the document, other than those imposed by security classification)

(x) Unlimited distribution
( ) Distribution limited to defence departments and defence contractors; further distribution only as approved
( ) Distribution limited to defence departments and Canadian defence contractors; further distribution only as approved
( ) Distribution limited to government departments and agencies; further distribution only as approved
( ) Distribution limited to defence departments; further distribution only as approved
( ) Other (please specify):

12. DOCUMENT ANNOUNCEMENT (any limitation to the bibliographic announcement of this document. This will normally correspond to the Document Availabilty (11). However, where further distribution (beyond the audience specified in 11) is possible, a wider announcement audience may be selected.)

UNCLASSIFIED

SECURITY CLASSIFICATION OF FORM

13. ABSTRACT (a brief and factual summary of the document. It may also appear elsewhere in the body of the document itself. It is highly desirable that the abstract of classified documents be unclassified. Each paragraph of the abstract shall begin with an indication of the security classification of the information in the paragraph (unless the document itself is unclassified) represented as (S), (C), (R), or (U). It is not necessary to include here abstracts in both official languages unless the text is bilingual).

A filter test system, the FTS 400, is capable of generating monodisperse aerosols in the size range 0.01 to 10 μm. This note describes operation of the main instrumental components, and notes precautions necessary in interpretation of results.

14. KEYWORDS, DESCRIPTORS or IDENTIFIERS (technically meaningful terms or short phrases that characterize a document and could be helpful in cataloguing the document. They should be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location may also be included. If possible, keywords should be selected from a published thesaurus. e.g. Thesaurus of Engineering and Scientific Terms (TEST) and that thesaurus identified. If it is not possible to select indexing terms which are Unclassified, the classification of each should be indicated as with the title.)

AEROSOLS
FILTERS
TESTING
EVALUATION
DIOCTYL PHTHALATE AEROSOLS
NaCl AEROSOLS
FTS 400

UNCLASSIFIED

SECURITY CLASSIFICATION OF FORM